

FORM PCT 1300  
REV. 5/93

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NO.  
PADEROV ET AL-1 (PCT)TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (If any) 09/710047

INTERNATIONAL APPLICATION NO.  
PCT/RU99/00336INTERNATIONAL FILING DATE  
SEPTEMBER 14, 1999PRIORITY DATE CLAIMED  
AUGUST 16, 1999

## TITLE OF INVENTION

METHOD FOR DEPOSITION OF WEAR RESISTANT COATINGS TO IMPROVE SERVICE LIFE OF  
COATED COMPONENTS

## APPLICANT(S) FOR DO/EO/US

ANATOLY NIKOLAEVICH PADEROV ET AL

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371 (f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau)
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has **NOT** expired.
  - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

## Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.  
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

PCT/ISA/210 - Int'l. Search Report (English)  
Small Entity Declaration  
6 Sheets of Formal Drawings

|   |              |  |  |
|---|--------------|--|--|
| APPLICATION NO. (if known, see 37 CFR 1.492(a)(1)-(5))  |              | INTERNATIONAL APPLICATION NO<br>PCT/RU99/00336                       | ATTORNEY'S DOCKET NO<br>PADEROV ET AL-1PCT |
| 09/700473   |              | CALCULATIONS   | PTO USE ONLY                               |
| The following fees are submitted:<br><b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b><br>Search Report has been prepared by the EPO or JPO.....\$860.00<br>International preliminary examination fee paid to USPTO (37 CFR 1.482).....\$710.00<br>Neither international preliminary examination fee paid (37 CFR 1.82) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....\$1000.00<br>International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4).....\$100<br><b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> |              | \$ 1,000.00  |  |
| Surcharge of \$130.00 for furnishing the oath or declaration later than 20 30 months from the earliest claimed priority date (37 CFR 1.492(e)).   |              |  |  |
| Claims  | Number Filed | Number Extra   | Rate                                       |
| Total Claims  | 15 - 20 =    | - 0 -  | X \$18.00                                  |
| Independent Claims  | 2 - 3 =      | 2  | X \$80.00                                  |
| Multiple dependent claim(s) (if applicable)   |              | + \$270.00   | \$   |
| <b>TOTAL OF ABOVE CALCULATIONS =</b>  |              | \$ 1,000.00  |  |
| Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).   |              | \$ 500.00  |  |
| <b>SUBTOTAL =</b>   |              | \$ 500.00  |  |
| Processing fee of \$130.00 for furnishing the English translation later than 20 30 months from the earliest claimed priority date (37 CFR 1.492(f)).  |              | \$   |  |
| <b>TOTAL NATIONAL FEE =</b>   |              | \$ 500.00  |  |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +  |              | See cover sheet attached to assign \$ to be charged to Deposit Acct. |  |
| <b>TOTAL FEES ENCLOSED =</b>  |              | \$ 500.00  |  |
|   |              | Amount to be: refunded   | \$   |
|   |              | charged  | \$   |

- a. ☒ A check in the amount of \$500.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 03-2468 in the amount of \$\_\_\_\_\_ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment, to Deposit Account No. 03-2468. A duplicate copy of this sheet is enclosed.

**(NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:  
 COLLARD & ROE, P.C.  
 1077 Northern Boulevard  
 Roslyn, New York 11576-1696  
 (516) 365-9802

*Edward R. Freedman*  
 Signature

Edward R. Freedman  
 Reg. No. 26,048

Express Mail No. **EL 621 999 650 US**

Date of Deposit **November 14, 2000**

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10, on the date indicated above, and is addressed to the Asst. Commissioner for Patents, Washington, D.C. 20231

*Lisa L. Vulpis*  
 Lisa L. Vulpis

PTO/PCT Rec'd 05 JUN 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS: ANATOLY N. PADEROV ET AL

SER. NO. 09/700,473

PCT NO.: PCT/RU99/00336 - Filed: 14 Sept. 1999

FOR: METHOD FOR DEPOSITION OF WEAR RESISTANT COATINGS  
TO IMPROVE SERVICE LIFE OF COATED COMPONENTS

PRELIMINARY AMENDMENT (to replace Preliminary  
Amendment filed on November 14, 2000)

Box PCT  
Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

Preliminary to the initial Office Action, please amend the  
above-identified application as follows:--

IN THE CLAIMS

Cancel claims 1-15, the original claims attached to the  
enclosed Response to Notification of Missing Requirements, and  
replace with new claims 16-30, attached hereto.

R E M A R K S

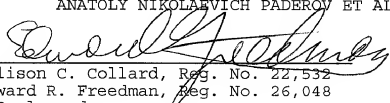
By this Preliminary Amendment, the changed claims submitted  
to the International Office are incorporated into the U.S. case.  
No new matter has been introduced. Entry of this amendment is  
respectfully requested.

This Preliminary Amendment, and the attached claims 16-30,  
are to replace the Preliminary Amendment and claims 15 to  
29 originally filed on November 14, 2000.

Respectfully submitted,

ANATOLY NIKOLAEVICH PADEROV ET AL

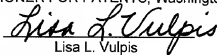
By

  
Allison C. Collard, Reg. No. 22,532  
Edward R. Freedman, Reg. No. 26,048

1077 Northern Boulevard

Roslyn, New York 11576  
(516) 365-9802  
Enclosure: New claims 16-30

I hereby certify that this correspondence is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 , and is addressed to:  
ASSISTANT COMMISSIONER FOR PATENTS, Washington, D.C. 20231, on June 6, 2001  
No. EL 769 392 699 US

  
Lisa L. Vulpis

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16. A method for depositing wear-resistant coatings on metal surfaces of machine components and articles, said method comprising the following steps:

- (i) providing an ion-plasma deposition chamber;
- (ii) locating said machine components or articles being treated inside said ion-plasma deposition chamber as an anode;
- (iii) locating in said chamber cathodes made from the Group IVA - VIA metals and/or alloys thereof;
- (iv) establishing in said chamber a gas atmosphere wherein the gas is selected from the group consisting of inert or non-inert gases and mixtures thereof;
- (v) effecting, whenever necessary, ion cleaning of surfaces of machine components or articles;
- (vi) effecting selective ion-plasma deposition of microlayers of a coating, wherein at least one microlayer (a) consists of said metals, mixtures thereof or substitution alloys, a second microlayer (b) consists of solutions of nonmetallic atoms of nitrogen, carbon, and boron in said metals, and a third microlayer (c) consists of chemical compounds of said metals with nonmetals in the form of nitrides, carbides, borides and mixtures thereof;
- (vii) subjecting one or more of said microlayers to treatment by implanting therein high energy non-metallic ions;
- (viii) cooling and unloading said machine components or articles from said chamber.

17. A method as defined in claim 16, CHARACTERIZED in that after having been cooled and unloaded said machine components or articles are subjected to vibromechanical treatment with micro-pellets.

18. A method as defined in claim 16, wherein said machine components or articles are made from titanium, titanium alloys, steels or nickel-based alloys.

19. A method as defined in claim 16, wherein said cathodes are selected from titanium alloys, steels or nickel-based alloys which after having been cooled form a composition similar to the base material of a machine component or article.

20. A method as defined in claim 16, wherein said plurality of microlayers is selected from the numbers 3-500, and said microlayers (a), (b), (c) alternate successively.

21. A method as defined in claim 19, wherein the thickness values of said microlayers (a), (b), (c) are in a ratio of 1.0:2.0:2.5.

22. A method as defined in claim 16, comprising preliminary deposition of a microlayer consisting of scandium, yttrium or other rare earth metal having a thickness of 0.02 to 0.08 micron before step (vi).

23. A method as defined in claim 16, wherein the reaction gas is nitrogen, acetylene, methane or diborane.

24. A method as defined in claim 16, wherein ion deposition is effected with ions of argon, or nitrogen, or carbon, or boron at an accelerating voltage of 10-50 kV, at a radiation dose of

1014 - 1018 ion/sq.cm and an energy of ions of  $5 \times 10^3$  -  $1 \times 10^5$  eV.

25. A method as defined in claim 16, wherein said ion-plasma deposition step (vi) comprises the steps of depositing:

(a) a scandium microlayer in argon atmosphere;

(b) a titanium microlayer in argon atmosphere;

(c) a microlayer comprising a solid solution of implanted nitrogen ions in titanium in an atmosphere comprising a mixture of nitrogen and argon;

(d) a microlayer comprising titanium nitride implanted with nitrogen ions in nitrogen atmosphere.

(e) a zirconium microlayer in argon atmosphere;

(f) a microlayer comprising a solid solution of implanted nitrogen ions in zirconium in an atmosphere comprising a mixture of nitrogen and argon;

(g) a microlayer comprising zirconium nitride implanted with nitrogen ions in nitrogen atmosphere; and

(h) the step of repeating said steps (b-g) to provide the required plurality of microlayers.

26. A method as defined in claim 16, wherein said deposition step (vi) comprises depositing:

(a) a first microlayer comprising alloys of titanium and zirconium in an inert gas atmosphere;

(b) a microlayer comprising alloys of titanium and zirconium implanted with nitrogen ions in an atmosphere of a mixture of said inert gas and nitrogen;

(c) a microlayer comprising titanium and zirconium nitrides implanted with nitrogen ions in nitrogen atmosphere;

(d) repeating said steps (a) - (c) to provide the required plurality of microlayers;

(e) ion deposition with argon ions of the deposited multilayer coating.

27. A method as defined in claim 26, wherein said deposition step (vi) comprising depositing:

(a) a microlayer of titanium and zirconium alloys in an inert gas;

(b) alloys of titanium and zirconium with boron in a mixture of an inert gas with diborane;

(c) titanium and zirconium borides implanted with boron;

(d) repeating said steps (a) - (b) to provide the required plurality of microlayers;

(e) ion implantation with argon the multilayer coating deposited.

28. A wear-resistant coating of metal surfaces, comprising deposited on said surfaces by ion-plasma deposition process at least one microlayer consisting of Group IVA - VIA metals, their mixture or substitution alloys, at least one microlayer consisting of solutions of nonmetal atoms of nitrogen, carbon, boron in said metals, at least one microlayer of chemical compounds of said metals with nonmetals in the form of nitrides, carbides, borides and mixtures thereof, implanted in at least one of any of said microlayers high-energy nonmetal ions selected from the group consisting of argon, nitrogen, carbon or boron ions.

29. Machine components and articles deposited by the method as claimed in claim 16.

30. Machine components and articles having a coating according to claim 28.



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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: ANATOLY NIKOLAEVICH PADEROV ET AL-1 (PCT)  
PCT No.: PCT/RU99/00336 FILED: SEPTEMBER 14, 1999  
TITLE: METHOD FOR DEPOSITION OF WEAR RESISTANT COATINGS  
TO IMPROVE SERVICE LIFE OF COATED COMPONENTS

PRELIMINARY AMENDMENT

**BOX PCT**

Ass't. Commissioner for Patents  
Washington, D.C. 20231

Dear Sir:

Preliminary to the initial Office Action, please amend the  
above-identified application as follows:

IN THE CLAIMS

Cancel claims 1-14 and replace with new claims 15-29, as  
follows: --

15. A method for depositing wear-resistant coatings on metal surfaces of machine components and articles, said method comprising the following steps:

- (i) providing an ion-plasma deposition chamber;
- (ii) locating said machine components or articles being treated inside said ion-plasma deposition chamber as an anode;
- (iii) locating in said chamber cathodes made from the Group IVA - VIA metals and/or alloys thereof;
- (iv) establishing in said chamber a gas atmosphere wherein the gas is selected from the group consisting of inert or non-inert gases and mixtures thereof;
- (v) effecting, whenever necessary, ion cleaning of surfaces of machine components or articles;
- (vi) effecting selective ion-plasma deposition of microlayers of a coating, wherein at least one microlayer (a) consists of said metals, mixtures thereof or substitution alloys, a second microlayer (b) consists of solutions of nonmetallic atoms of nitrogen, carbon, and boron in said metals, and a third microlayer (c) consists of chemical compounds of said metals with nonmetals in the form of nitrides, carbides, borides and mixtures thereof;
- (vii) subjecting one or more of said microlayers to treatment by implanting thereinto high energy non-metallic ions;
- (viii) cooling and unloading said machine components or articles from said chamber.

16. A method as defined in claim 15, CHARACTERIZED in that after having been cooled and unloaded said machine components or articles are subjected to vibromechanical treatment with micro-pellets.

17. A method as defined in claim 15, wherein said machine components or articles are made from titanium, titanium alloys, steels or nickel-based alloys.

18. A method as defined in claim 15, wherein said cathodes are selected from titanium alloys, steels or nickel-based alloys which after having been cooled form a composition similar to the base material of a machine component or article.

19. A method as defined in claim 15, wherein said plurality of microlayers is selected from the numbers 3-500, and said microlayers (a), (b), (c) alternate successively.

20. A method as defined in claim 18, wherein the thickness values of said microlayers (a), (b), (c) are in a ratio of 1.0:2.0:2.5.

21. A method as defined in claim 15, comprising preliminary deposition of a microlayer consisting of scandium, yttrium or other rare earth metal having a thickness of 0.02 to 0.08 micron before step (vi).

22. A method as defined in claim 15, wherein the reaction gas is nitrogen, acetylene, methane or diborane.

23. A method as defined in claim 15, wherein ion deposition is effected with ions of argon, or nitrogen, or carbon, or boron at an accelerating voltage of 10-50 kV, at a radiation dose of  $10^{14} - 10^{18}$  ion/sq.cm and an energy of ions of  $5 \times 10^3 - 1 \times 10^5$  eV.

24. A method as defined in claim 15, wherein said ion-plasma deposition step (vi) comprises the steps of depositing:

- (a) a scandium microlayer in argon atmosphere;
- (b) a titanium microlayer in argon atmosphere;

(c) a microlayer comprising a solid solution of implanted nitrogen ions in titanium in an atmosphere comprising a mixture of nitrogen and argon;

(d) a microlayer comprising titanium nitride implanted with nitrogen ions in nitrogen atmosphere.

(e) a zirconium microlayer in argon atmosphere;

(f) a microlayer comprising a solid solution of implanted nitrogen ions in zirconium in an atmosphere comprising a mixture of nitrogen and argon;

(g) a microlayer comprising zirconium nitride implanted with nitrogen ions in nitrogen atmosphere; and

(h) the step of repeating said steps (b-g) to provide the required plurality of microlayers.

25. A method as defined in claim 15, wherein said deposition step (vi) comprises depositing:

(a) a first microlayer comprising alloys of titanium and zirconium in an inert gas atmosphere;

(b) a microlayer comprising alloys of titanium and zirconium implanted with nitrogen ions in an atmosphere of a mixture of said inert gas and nitrogen;

(c) a microlayer comprising titanium and zirconium nitrides implanted with nitrogen ions in nitrogen atmosphere;

(d) repeating said steps (a) - (c) to provide the required plurality of microlayers;

(e) ion deposition with argon ions of the deposited multilayer coating.

26. A method as defined in claim 25, wherein said deposition step (vi) comprising depositing:

(a) a microlayer of titanium and zirconium alloys in an inert gas;

(b) alloys of titanium and zirconium with boron in a mixture of an inert gas with diborane;

(c) titanium and zirconium borides implanted with boron;

(d) repeating said steps (a) - (b) to provide the required plurality of microlayers;

(e) ion implantation with argon the multilayer coating deposited.

27. A wear-resistant coating of metal surfaces, comprising deposited on said surfaces by ion-plasma deposition process at least one microlayer consisting of Group IVA - VIA metals, their mixture or substitution alloys, at least one microlayer consisting of solutions of nonmetal atoms of nitrogen, carbon, boron in said metals, at least one microlayer of chemical compounds of said metals with nonmetals in the form of nitrides, carbides, borides and mixtures thereof, implanted in at least one of any of said microlayers high-energy nonmetal ions selected from the group consisting of argon, nitrogen, carbon or boron ions.

28. Machine components and articles deposited by the method as claimed in claim 15.

29. Machine components and articles having a coating according to claim 27.

REMARKS

By this Preliminary Amendment, the changes submitted to the International Office are incorporated into the U.S. case. In particular, original claims 1-14 have been replaced by new claims 15-29. Entry of this amendment is respectfully requested. No new matter has been introduced.

Respectfully submitted,  
ANATOLY NIKOLAEVICH PADEROV ET AL

By: 

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Lisa L. Vulpis

# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS OR PATENTEE: Anatoly N. PADEROV ET AL

PCT NO.: PCT/RU99/00336

PCT FILED: Sept. 14, 1999

GROUP:

TITLE: METHOD FOR DEPOSITION OF WEAR RESISTANT COATINGS TO IMPROVE ...

## SMALL ENTITY DECLARATION

☒ [ X ] FOR INDEPENDENT INVENTOR(S)

As a below-named inventor, I hereby declare that I am an independent inventor who (1) has not assigned, granted, conveyed, or licensed, and (2) is under no obligation under contract or law, to assign, grant, convey, or license, any rights in the invention, to any person who could not likewise be classified as an independent inventor if that person had made the invention, or to any concern which would not qualify as a small business concern or a nonprofit organization, as defined in 37 CFR 1.9(c).

☐ [ ] FOR SMALL BUSINESS CONCERN

I hereby declare that \_\_\_\_\_ is a business concern which qualifies as a small business concern as defined in §1.9(d) - namely, (1) whose number of employees, including those of its affiliates, does not exceed 500 persons; and (2) which has not assigned, granted, conveyed, or licensed, and is under no obligation under contract or law to assign, grant, convey, or license, any rights in the invention to any person who could not be classified as an independent inventor if that person had made the invention, or to any concern which would not qualify as a small business concern or a nonprofit organization under this section; and that the exclusive rights to the invention have been conveyed to and remain with the above-identified small business concern.

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5     Method for Deposition of Wear Resistant Coatings to Improve Service Life of  
          Coated Components

Field of the Invention

10       This invention relates to metallurgy and machine building fields and more specifically to development of methods that improve service life and durability of machine components; to repair of components and reconstitution of their properties; and particularly to gas turbine blades and vanes; and primarily to coatings applied to metal surfaces of aircraft engine compressor blades and vanes.

15                   Background of the Invention

          Frequently, aircraft and helicopters equipped with gas-turbine engines have to operate under conditions of considerable dust content in the air flow and high humidity of sea environment with aggressive elements of corrosive effects. These operation conditions result in abrasion-caused erosion and corrosion of aircraft parts, particularly the compressor blades. Under these conditions, geometry of blades is distorted, operating performances are deteriorated, fuel consumption increases, and engine maintenance and repair expenses grow considerably. The said deteriorating processes can not be efficiently avoided by installation of dust protective devices.

          Eroded blades and vanes are generally restored by edge profile polishing or are replaced with new blades and vanes. Such blades and vanes are made of titanium-based alloys or high-alloy steels, which are expensive and difficult to process, so engine repair entails great expense.

30       U.S. Patent No.4,904,542, issued February 27, 1990, reissued under Re.34,173 on February 2, 1993, to Midwest Research Technologies Inc. describes a coating formed of a plurality of alternating layers of metallic and ceramic materials. The two materials selected for the layers have complementary wear resistant characteristics such that one is relatively ductile and the other is relatively hard and brittle.

35       Preferably radio-frequency sputtering is employed to deposit the coating, since it does not produce excessive heating which could negate any prior heat treatment of the substrate onto which the coating is deposited.



Also known are RU Patents No.2,061,090 BI No. 15, 1996 and No.2,106,429 BI No. 7, 1998, that describe methods of multi-layer coating deposition on parts and tools, including transition metal coatings. Zirconium is offered as an adhesive bondcoat that is applied to the substrate before the coating; or there is an alternative method of applying metal oxides between the metal layers.

A deposition technique is also known to produce thin films of CN<sub>x</sub> with implantation of nitrogen ions from plasma. U.S. Patent No. 5,580,429 issued December 3, 1996, to Northeastern University describes cathodic/anodic vacuum arc sources with a plasma ion implantation deposition system for depositing high quality thin film coatings on substrates. Both cathodic and anodic vacuum arc deposition sources, CAVAD, are used to create a plasma vapor from solid materials composing the cathode and/or anode in the cathodic and/or anodic arc, respectively. Gases, e.g., hydrogen or nitrogen can be in the deposited films by creating a background plasma of the desired gas using either RF energy, thermionic emission, or consequential ionization of the gas passing through the arc or around the substrate. Highly negative pulses are applied to the substrate to extract the ions and provide them with the appropriate energy to interact with the other species in the thin film formation on the substrate to form the desired films. The substrate is bombarded with ionized particles to form carbon nitrides with variable [N]/[C] ratios.

RU Patent No.2,062,818 issued June 27, 1996, describes deposition of metal-containing coatings on large substrates in vacuum. The method includes inert gas ion beam cleaning of the substrate and metal-coating deposition by cathodic sputtering in the inert gas discharge when the substrate is bombarded with the inert gas ion beam that is formed by an accelerator of closed-type drift of electrons at an inert gas ion energy of 50-150 eV. Technically, this method is the closest one to the present invention.

However, the aforesaid U.S.P. 4,904,542, U.S.P. 5,580,429 and R.U. 2,062,818, R.U. 2,061,090, and R.U. 2,106,429 do not fully cover the problems of durability and wear resistance, especially as far as aircraft engine blade airfoil surfaces are concerned, which must meet various specific requirements to their wear and corrosion resistance properties and at the same time retain a certain level of their mechanical and, particularly, fatigue characteristics.

Therefore, there is a need to provide improved erosion and corrosion resistance and, as a result, improved reliability and durability of components of

- 5 various machines, tools and equipment, especially gas turbine engine compressor blades and vanes. That is proposed to be achieved by vacuum plasma technology involving ion implantation.

### Summary of the Invention

10

It is an object of the present invention to provide a technique of coating deposition on metal surfaces, particularly on components of machines, steam and gas turbines, and even more specifically on aircraft engine compressor blades and vanes, that will ensure improved erosion and corrosion resistance and retain the sufficient

15 level of mechanical properties, primarily, fatigue characteristics.

It is a further object to restore the metallic surface of an eroded or corroded metal substrate, particularly the profile surface of a working blade of a gas turbine engine compressor to its original geometric shape and profile parameters.

- 20 To achieve the aforementioned objects, a coating is deposited that consists of at least three or four microlayers with certain thickness and compositions. By the term "microlayer", in this specification and claims, is meant a layer of pure metal, multiple-component substitution or interstitial metal alloy with non-metal atoms, or interstitial phases based on the said metals, i.e. the metal carbides, nitrides, borides, or complex compounds of the said phases, e.g., carbonitrides, carborides, etc..

- 25 The said coating is produced by means of ion plasma deposition; in the preferred embodiments, the said coating consists of a special microlayer (hereinafter referred to as "submicrolayer"); the said submicrolayer is a rare earth metal, particularly scandium, yttrium or lanthanum and lanthanoids; the said coating also comprises a plurality of microlayers wherein each of said microlayers comprises a
- 30 material selected from the group consisting of the Group IVA-VIA (Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W) or alloys thereof, interstitial solid solutions of elements (carbon, nitrogen, boron), nitrides, carbides, or borides of metals, wherein one or more of said microlayers has been subjected to high energy non-metallic (argon, nitrogen, carbon, boron) ion deposition.

- 35 The said microlayers of metals or said alloys or metal/non-metal compounds deposited by means of deposition of ions or neutral particles under an appropriate inert gas or non-inert gas, such as nitrogen, methane, acetylene, diborane, should be deposited to a desired thickness, preferably 0.1-10 microns.

5           The microlayer may be an essentially discrete layer distinct from the adjacent substrate or microlayers; or it may be a mixture therewith. Each of the microlayers may comprise a pure metal or an alloy thereof as prepared, for example, if more than one metallic cathode are simultaneously activated within the chamber or the cathodes are made of alloys. The order of the plurality of microlayers can be selected by opting  
 10       between corresponding gas atmospheres in the working chamber and by activating the appropriate cathode(s). The number and order of microlayers constituting the full coating and the inert or non-inert gas ions deposition can be selected depending on the specific requirements determined by the desired performances of machine parts or the whole machine. For example, it is essential that guide blades of the aircraft engine  
 15       compressor had very hard and wear resistant surfaces, and at the same time fatigue characteristics of the substrate alloy would not play a restrictive role, since such blades are not subjected to high fatigue. On the contrary, working blades of the compressor are very sensitive to fatigue conditions as such blades have to operate under considerable fatigue stresses. Therefore, coatings designed for guide and  
 20       working blades differ in their thickness and number of microlayers.

The method claimed involves deposition of, at least, three functional microlayers:

1   - a damping, corrosion-resistant microlayer of a rare earth metal from the Groups IVA - VIA or a replacement alloy based on said metals, deposited in inert gas  
 25       atmosphere to the desired thickness, preferably 0.02 - 5 microns, that provides relaxation of erosion-caused stresses between solid layers and protects from corrosion-aggressive agents of media;

2   - a reinforcing microlayer consisting of interstitial solid solutions of nitrogen, boron, carbon in transition metals of the second layer, deposited to the  
 30       desired thickness, preferably 0.04 - 10 microns, in a non-inert gas (nitrogen, diborane, methane, or acetylene, respectively, at a partial pressure of said gases  $0.05 - 5 \times 10^{-1} \text{ Pa}$ ) atmosphere, that provides a gradual transition to a high strength layer;

3   - a wear-resistant, high strength microlayer consisting of interstitial phases such as nitrides, borides, carbides or complex compounds thereof based on said  
 35       transition metals, deposited to the desired thickness, preferably 0.1 - 12.5 microns, in corresponding non-inert gas atmospheres at a partial pressure of  $0.1 - 5 \times 10^{-1} \text{ Pa}$ , that provides resistance to erosion effects of abrasive particles.

The deposition of the aforementioned functional microlayers is carried out by activating the appropriate cathode made of a pure metal or a multiple-component alloy, by selecting the necessary partial pressure and composition of the gas atmosphere, and by controlling the appropriate time of deposition as required.

Fig. 1 shows an exemplary microstructure of the claimed coating on an aircraft engine compressor blade of titanium alloy.

Simultaneously, one or more microlayers is subjected to non-metallic (argon, nitrogen, carbon or boron) ion treatment by means of an ion implantor; it is important that such treatment must be carried out directly in the working chamber of the ion-plasma device, simultaneously with or immediately following the deposition process.

The ion treatment is carried out with ions at  $5 \times 10^3 - 1 \times 10^5$  eV and radiation dose of  $5 \times 10^{13} - 1 \times 10^{18}$  ion/cm<sup>2</sup>.

The energy of these implanted ions is considerably higher than the energy of ions formed in the deposition chamber. These ions penetrate deep into the crystal lattice of the deposited metals or interstitial phases and cause changes in the interstitial element concentration, bring about formation of solid solutions and superstructural, non-stoichiometric compounds, and result in submicrostructure and strain modifications. All these result in improved adhesion strength and higher resistance of the coatings to erosion wear. Under the effect of such ion treatment, local temperature peaks may occur followed by rapid cooling of these surface localities, that results in the improvement of strength and tribological properties of the deposited microlayers.

Fig. 2 shows an exemplary X-ray pattern obtained from coatings deposited under various ion implant treatments in the deposition chamber; and Fig. 3 shows the results of investigation by means of the Rutherford back scattering.

It is preferable to use a high energy pulsed ion source in order to reduce risks of overheating and temperature warping of a machine part under ion plasma deposition, which is especially important for aircraft engine compressor blades. Such source produces ions that have the energy high enough for the ions to be implanted into the crystal lattice of deposited phase and to create high-tensile compounds. The following rapid cooling of affected zones prevents the bulk material from overheating, causes the surface substructure to become finer and brings about nanocrystalline or amorphous structures in the surface microlayers.

The ion treatment improves, not only the resistance to erosion and corrosion, but also the endurance limit of machine components, especially under many-cycled fatigue conditions. The improvements are achieved due to the compressing strains generated in the interfacial boundaries and stable defects in the structure caused by the formation of fine precipitates of multiple-component metal/non-metal compounds of variable valence.

However, such complicated processes of multiple-layer coating deposition and ion implanted treatment may induce elevated internal stresses in the surface layers of machine components. In order that the stress distribution be favorable, it is necessary to carry out an additional treatment of the coated parts immediately after the deposition process. After the coated parts are unloaded from the deposition chamber, they are subjected to vibromechanical treatment with micro-pellets.

Therefore, the method claimed includes the following steps:

- (a) Preparing the surface for ion-plasma deposition.
- (b) Installing cathodes made of metals and alloys to be deposited.
- (c) Placing workpieces or substrates into the ion plasma deposition chamber equipped with an ion implantor.
- (d) Ion beam cleaning of the surface.
- (e) Ion plasma depositing of coating comprising a plurality of microlayers with the required compositions and gas pressures in the deposition chamber.
- (f) High-energy ion treatment of one or more microlayers during their deposition or after the coating has been deposited.
- (g) Cooling and unloading the workpieces.
- (h) Vibro-mechanical treatment according to the preset regime.

#### Brief Description of the Drawings and Photographs

Fig. 1 Microstructure of the wear-resistant coating on an aircraft engine blade of titanium-based alloy, x500x2;

Fig. 2 X-ray diffraction patterns from coatings with various implanted ions and regimes;

Fig. 3 Results of investigation of coating by means of Rutherford back scattering;

Fig. 4 Erosion values represented by weight loss in testing conducted on gas-turbine engine compressor blades;

Fig. 5 Erosion values represented by chord wear in testing conducted on gas-turbine compressor blades;

- 5 Fig. 6 Fatigue test results obtained on test specimens and compressor blades with and without coating.

### Detailed Description of Preferred Embodiments

10 In practice, ion-plasma deposition of metal ions is carried out following the general principles of ion plasma deposition in a low pressure chamber from the corresponding metal cathode in an inert gas atmosphere, for instance, in argon or, in order to produce a nitride metal coating, in nitrogen, with a considerable potential difference between the hot cathode and a workpiece that plays a role of the anode.

15 The coatings listed in Table 1 were prepared as follows. Ion-plasma deposition and high-energy ion treatment were conducted in HHB-6.6 type of equipment with an ion implantor of "Pulsar" type, or an implantor with a non-heated cathode, equipped with high accuracy optical pyrometers and inert/non-inert gas feeding systems monitored to feed the gas atmosphere into the ion plasma deposition chamber and implantor.

20 Titanium, steel or nickel-based alloy aircraft engine compressor blades were first treated with high energy ion argon plasma at a potential difference up to 1,500 V between titanium cathode and the blades, to clean the blade airfoil surface from any solid, liquid or adsorbed gas impurities.

25 Let us consider in more detail the coating deposition embodiment designated as No. 8. After the blades of BT16 alloy were treated with abrasive liquid, washed and dried, the blades were then placed into the ion-plasma deposition chamber where they were subjected to ion cleaning in inert gas atmosphere and then they were coated with a plurality of microlayers, starting with the scandium submicrolayer.

30 The scandium submicrolayer of 0.3 - 0.8 micron in thickness was deposited on the blade airfoil surface, at a scandium cathode heating current of 10 - 200 A to provide a temperature of 200 - 400°C and a potential difference of 100 - 1,000 V between the blades and the scandium cathode. Titanium cathodes and zirconium cathode were not heated this time. The stage took approximately 2 minutes and the blades were rotated at 2.5 rpm. A titanium microlayer having a thickness of 0.6 - 1.6 microns was then deposited by inactivating the scandium cathode by switching off its source of current and heating the titanium cathode by applying a current of 20 - 200 A and a potential difference of 100 - 800 V between the anode and the blades under the

5 argon atmosphere. A temperature of the cathode up to 700°C was achieved over this stage. Then a titanium microlayer having a thickness of 1-2 microns was deposited under a nitrogen and argon atmosphere in the working chamber. Then pressure was increased and a microlayer of titanium nitride was formed of a thickness approximately 2 - 4 microns. The temperature of blades was maintained within 480 - 10 550°C over the whole deposition process to prevent any phase transformation in the bulk material.

The titanium cathode was inactivated and zirconium cathode heated to the same temperature by the same current density and potential difference as for the preceding titanium deposition step. During the deposition of titanium nitride and 15 zirconium nitride there was nitrogen implantation carried out. The aforesaid deposition steps were repeated several times in the same sequence in order to obtain the desired thickness of the coating.

In alternative embodiments the foregoing titanium and zirconium ion deposition steps may be repeated, substituted or interchanged with titanium nitride 20 and/or zirconium nitride ion deposition steps carried out under a nitrogen atmosphere. Interchanging of different microlayers is provided by the alternating heating of the titanium or zirconium cathodes under an argon or nitrogen atmosphere. An example is shown in Fig. 3 of changes in composition of various microlayers of the coating deposited following the claimed method and analyzed by means of the Rutherford 25 back scattering. Clearly, a desired total thickness of coating can be obtained from a plurality of microlayers, preferably 3 - 20.

In preferred embodiments, each or some of the microlayers of the full coating at different stages in its preparation may be subjected to high energy ions of argon, nitrogen, carbon, or boron as selected by control of the atmosphere in the ion 30 implantor under a potential difference between the ion-implantor electrode and the blades of 10 - 50 kV.

Ion implantors are well-known in the art.

In the present embodiment, the ion implantor of "Pulsar" type is provided with a low pressure arc between a screened cathode spot and a widened anode part of the 35 discharge. The arc provides a high current of non-metallic ions of argon or a non-inert gas medium injected into the implantor from the developed emission surface of the anode plasma. Cathode ion emission is negligible since the cathode is not heated. Further, on screening of the cathode spot prevents its interference with the anode

- 5 plasma and lowers contamination of the gas-discharging plasma by metallic ions. Accordingly, only ions from the arc anode plasma enter the ion optical system which forms the beam of high-energy ions. The plasma contains less than 0.1% of metal ions.

10 In the process according to the invention the following process parameters were used:

|   |                                      |
|---|--------------------------------------|
| Accelerating voltage  | up to 50 kV                          |
| Ion beam amperage: in impulse   | 1 A                                  |
| average   | 50 mA                                |
| Beam cross-section: wide beam   | 150 cm <sup>2</sup>                  |
| 15 convergent beam  | 5 cm <sup>2</sup>                    |
| Impulse duration  | 1×10 <sup>-3</sup> sec <sup>-1</sup> |
| Repetition rate   | 1 - 50 sec <sup>-1</sup>             |
| Ion types - any gas ions, including ions of chemically active and inert gases |                                      |

- 20 A beam of nitrogen or boron or carbon or argon ions, emanating from an emitting electrode is accelerated through an accelerating electrode and an outlet electrode to intermittently impinge, as desired, on the individual microlayers of the substrate.

25 Table 1 gives the structural order and compositions of several examples of coated substrates according to the invention.

Table 1

| Example No. | Substrate material | No. of microlayers in the coating | Total coating thickness (microns) | Material and order of coating                 |
|-------------|--------------------|-----------------------------------|-----------------------------------|---|
| 1           | Titanium alloy     | 7                                 | 12-16                             | Sc,Ti+Zr,(Ti,Zr(N)),<br>Zr,(Zr(N)),<br>ZrN+Ar |
| 2           | Stainless steel    | 9                                 | 10-12                             | Sc,Ti+Zr,(Ti,Zr(N)),Ti<br>N+N,ZrN+N*          |
| 3           | Titanium alloy     | 8                                 | 6-8                               | Ti,(Ti(N)),TiN,<br>ZrN+N*                     |
| 4           | Nickel-based alloy | 4                                 | 4-6                               | Y,Ti,(Ti(N)), TiN+Ar                          |
| 5           | Titanium alloy     | 17                                | 14-18                             | Sc,Ti,(Ti(N)),<br>TiN+N,Ti,+N,Zr,             |



|    |                    |    |       |   |
|----|--------------------|----|-------|---|
|    |                    |    |       | (Zr(N)), TiN, ZrN+N*  |
| 6  | Nickel-based alloy | 19 | 18-22 | REM, Zr, (Zr(N)),<br>ZrN, +Ar, Zr, (Zr(N))ZrN<br>+Ar, Ti, (Ti(N)),<br>TiN+Ar*   |
| 7  | Titanium alloy     | 46 | 18-22 | Sc, Ti, (Ti(N)), TiN, +N,<br>Zr, (Zr(N)), ZrN, Ti,<br>(Ti(N)), TiN+N,<br>Ti, (Ti(N)), TiN+N,<br>Zr, (Zr(N)), ZrN                |
| 8  | Ti-6Al-4V alloy    | 19 | 14-18 | Sc, Ti, (Ti(N)),<br>TiN, +N, Zr, (Zr(N)),<br>ZrN, +N, Ti,<br>(Ti(N)), TiN*  |
| 9  | Titanium alloy     | 25 | 28-32 | Sc, Ti, (Ti(N)), TiN+NZr<br>N+TiN, Zr+N, Ti,<br>(Ti(N)), TiN+Ar   |
| 10 | Titanium alloy     | 40 | 16-20 | Cr, (Cr(C)), Cr <sub>3</sub> C <sub>2</sub> ,<br>ZrC+C, Zr, (Zr(C)),<br>ZrC+C, ZrC, Zr,<br>Cr <sub>3</sub> C <sub>2</sub> +ZrC* |
| 11 | Titanium alloy     | 57 | 14-18 | Y, Ti, (Ti(C)), TiC+C, Ti, (Ti(C)), ZrC+C,<br>Zr+C, Ti+C, ZrC, Ti,<br>(Ti(C)), TiC+C, ZrC+Ti<br>C, ZrC*                         |
| 12 | Titanium alloy     | 49 | 14-20 | Y, Zr, (Zr(C)), ZrC+CZr, (Zr(C)), TiC+C, Ti<br>(Ti(C)), TiC+C, Zr,<br>(Zr(C)), ZrC*   |

5

Note: +C, +N, +Ar - denote carbon, nitrogen or argon ion implant treatment respectively;

(Ti(N)), (Zr(N)), (Ti(C)), (Zr(C)), etc. - the microlayers comprising interstitial solid solutions of nitrogen and carbon in the corresponding metal;

10 \* - the microlayers are deposited in the specified order several times to obtain coating of desirable thickness;

Ti+Zr - the microlayer comprising of the mixture of the metals specified.

5

Fig.4 and Fig. 5 show erosion resistance of complex coatings comprising a plurality of microlayers produced, according to the invention, of zirconium, titanium and nitrides thereof, and subjected to erosion tests on Ti-6Al-4V alloy compressor blades with implanted nitrogen ions as compared to the non-coated blades. The Figures also specify the testing conditions.

Specified below is wear resistance test results, of coating specimens having the same number of microlayers and a total thickness of 12 - 16 microns with and without nitrogen or carbon implanted ions.

1. BT8 alloy (Ti-6Al-3.5Mo-0.5Zr) without coating = 1.0
2. Coating on BT8 alloy - Sc-Ti-(Ti(N))-TiN-Zr-(Zr(N))-ZrN = 0.12
3. Coating on BT8 alloy - Sc-Cr-(Cr(C)) - Cr<sub>2</sub>C<sub>3</sub>-(Zr(N))-ZrC = 0.26
4. Coating on BT8 alloy - Sc-Ti-(Ti(N))-TiN+N-Zr-(Zr(N))+N = 0.014
5. Coating on BT8 alloy - Sc-Cr-(Cr(C))- Cr<sub>2</sub>C<sub>3</sub>+C-Zr-(Zr(C))+C = 0.037

#### Testing conditions:

- Velocity of the air-abrasive flow - 120 m/sec
- Temperature - room temperature
- Abrasive material - quartz sand, of 10 microns granularity
- Abrasive material quantity - 10 kg
- Attack angle - 20°
- Specimens 4 and 5 are with implanted nitrogen and carbon ions, respectively
- Wear resistance was measured by weight loss as compared with non-coated specimens.

Corrosion tests

Coated as per the invention and non-coated steel and titanium blades were subjected to corrosion tests by the method as follows.

10 The titanium blades were heated at 300°C and the stainless steel blades at 420°C in a chamber for 1 hour. The blades were subsequently cooled in a 3% sodium chloride solution, held in a humid chamber for 23 hours and the cycle was repeated 10 times. These tests were meant to simulate 2 years of fan blade operation under conditions of humid tropical climate. Corrosion resistance evaluation was made by visual examination of the blades and vanes after each cycle and by determination of  
15 the change in mass during and after the corrosion tests.

The results showed that non-coated blades had a typical change in mass of 1.3g/m, in contrast to the coated blades which had no corrosion as measured visually neither any change in weight.

Another test was conducted in a chamber at 350°C in 3% sodium chloride  
20 solution vapors for 3 days. This test was meant to estimate pitting corrosion on the leading edge of the blades (to simulate corrosion conditions at moorings), and then the data obtained were averaged over 8 blades (see below):

1. Non-coated blades - over 20 pitting marks in the leading edge
2. Titanium nitride coated blades - 9-12 pitting marks
- 25 3. Blades coated with a plurality of microlayers and argon implanted according to the invention - 1 -3 pitting marks.

The comparison tests conducted on non-coated blades and the blades coated according to the invention have shown that the fatigue strength of the coated blades  
30 does not deteriorate and thus provides a high level of durability and high endurance limit (Fig.6). The fatigue test parameters were corresponding to the actual operating conditions of aircraft engine performance.

After the laboratory tests had been conducted, natural scale tests were performed on real engines, with abrasive material particles of 100 - 200 microns at the  
35 sand consumption rate of 1.2 kg/hour. These tests have also demonstrated a considerable improvement in erosion resistance of the aircraft compressor guide blades and rotor under severe conditions as compared to the non-coated blades. Fig.7 shows the surface of the blades. Therefore, the results obtained can be used to

- 5     develop a repair technology designed for improving durability of new blades of an aircraft engine.

Industrial applicability

- 10             The invention claimed can be used to improve durability and endurance limit of machine components by depositing wear and corrosion resistant coating on the component parts used in a variety of machine building industries, and to repair and restore component parts worn in operation. Particularly, positive results have been obtained for restoring worn-out compressor blades and for improving aircraft engine
- 15     compressor's durability and service life. Technologies based on the claimed invention can also be utilized in other industries, such as manufacture of consumer goods, domestic appliances, and sporting equipment.

We claim

1. A method for deposition of coating, comprising a plurality of microlayers, wherein each of said microlayers comprises a material selected from the group consisting of the Group IVA - VIA metals and alloys thereof, or interstitial phases based thereon, wherein one or more of said microlayers is subjected to high energy non-metallic ion deposition; also a method for improvement of durability of machine components by said coating deposition followed by vibromechanical treatment with micro-pellets; said method including the steps of:

- (i) locating said machine components as an anode inside an ion-plasma deposition chamber;
- (ii) locating a cathode comprising a metal selected from said metals and alloys thereof within said chamber;
- (iii) feeding said chamber with a gas atmosphere wherein the gas is selected from the group consisting of inert or non-inert gases and mixtures thereof;
- (iv) ion cleaning of surfaces to be coated;
- (v) effecting selective ion-plasma deposition of said microlayers comprising said metals or alloys thereof or interstitial phases based thereon;
- (vi) subjecting one or more of said microlayers to high energy non-metallic ion deposition;
- (vii) cooling and unloading said machine components or articles from said chamber followed by vibromechanical treatment with micro-pellets.

2. A method as defined in claim 1, wherein said machine components or articles are selected from those made of materials comprising titanium, titanium alloys, steels or nickel-based alloys.

3. A method as defined in claim 1, wherein said cathodes are selected from those made of materials comprising titanium, titanium alloys, steels or nickel-based alloys that after deposition provide composition similar to the base material of the components or articles.

4. A method as defined in claim 1, wherein said plurality of microlayers is selected from the numbers 3-500, and increasing the number of microlayers is effected through alternating said metals, alloys of said metals or interstitial phases based thereon.

5. A method as defined in claim 4, wherein said microlayers are deposited at a thickness proportion of (0.02-0.5): (0.04-10): (0.1-12.5), in which the first three layers are deposited at a thickness proportion of 1 : 2 : 2.5.

6. A method as defined in claim 1, wherein said ion plasma deposition comprises depositing a microlayer consisting of scandium, yttrium or other rare earth metal to a thickness selected from 0.02 - 0.08 micron following said ion cleaning according to step (iv) before the step (v).

7. A method as defined in claim 1, wherein said gas is selected from the group consisting of nitrogen, acetylene, methane and diborane.

8. A method as defined in claim 7, wherein formation of layers of the alloys and interstitial phases is provided by the ratio of  $(0,005-0,2 : 0,01-7,0)10^{-1}$  Pa between partial pressures of the reaction gases.

9. A method as defined in claim 1, wherein said high-energy non-metallic ion deposition is effected with said non-metallic ions selected from the group consisting of argon, nitrogen, carbon, and boron at an accelerating voltage selected from 10-50 kV, at a radiation dose of  $10^{14} - 10^{18}$  ion/cm<sup>2</sup> and an energy of ions of  $5 \times 10^3 - 1 \times 10^5$  eV.

10. A method as defined in claim 1, wherein said vibromechanical treatment is effected not later than 10-60 min upon coating deposition, with micro-pellets of 0.5 - 5.0 mm in diameter at an amplitude of 2 - 8 mm.

11. A method as defined in claim 1, wherein said ion-plasma deposition step (v) comprises the steps of selectively depositing:

- (a) a scandium submicrolayer in argon atmosphere;
- (b) a titanium microlayer in argon atmosphere;
- (c) a microlayer comprising a solid solution of implanted nitrogen ions in titanium in an atmosphere comprising a mixture of nitrogen and argon;
- (d) a microlayer comprising titanium nitride implanted with nitrogen ions in nitrogen atmosphere;
- (e) a zirconium microlayer in argon;
- (f) a microlayer comprising a solid solution of implanted nitrogen ions in zirconium in an atmosphere comprising a mixture of nitrogen and argon;
- (g) a microlayer comprising zirconium nitride implanted with nitrogen ions in nitrogen atmosphere; and
- (h) the step of repeating said steps (b-g) to provide the required plurality of microlayers.

12. A method as defined in claim 1, wherein said deposition step (v) comprises selectively depositing:

- (a) a first microlayer comprising alloys of titanium and zirconium in said inert gas atmosphere;
- (b) a microlayer comprising said alloys of titanium and zirconium implanted with nitrogen ions in an atmosphere of a mixture of said inert gas and nitrogen;
- (c) a microlayer comprising titanium and zirconium nitrides implanted with nitrogen ions in nitrogen atmosphere;
- (d) repeating said steps (a - c) to provide said plurality of microlayers;
- (e) high energy ion deposition with argon ions of the resultant coating comprising said plurality of microlayers.

13. A method as defined in claim 12, wherein said step (b) comprises depositing titanium and zirconium alloys with carbon in a mixture of an inert gas with acetylene or methane, and wherein said step (c) comprises depositing titanium and zirconium carbides implanted with carbon ions in an atmosphere of acetylene or methane.

14. A method as defined in claim 12, wherein said step (b) comprises depositing titanium and zirconium alloys with boron in a mixture of an inert gas with diborane, and wherein said step (c) comprises depositing titanium and zirconium borides implanted with boron ions.

15. A method as defined in claim 1, wherein said machine components are gas turbine compressor blades and vanes or parts thereof.

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ABSTRACT OF THE DISCLOSURE

This invention relates to metallurgy and machine building, more specifically to the development of a method that improves service life, durability and repair of machine components by applying coatings to working surfaces followed by special treatment of the surfaces.

The essence of the invention is deposition of erosion and corrosion resistant coatings on machine components, that comprises a plurality of microlayers wherein each of the microlayers comprises one or more elements selected from the transition metal group, solid solutions or interstitial phases based thereon, and wherein one or more of the microlayers is subjected to high energy non-metallic ion deposition that causes changes in structure and composition of the deposited microlayer thus improving performance characteristics. After the full coating has been deposited, a vibromechanical treatment with micro-pellets is applied to the surface of machine components, that improves distribution of residual stresses.

The method makes it possible to deposit coatings having high resistance to wear and corrosion, and having a sufficient level of fatigue strength of machine components, primarily gas-turbine compressor blades and vanes.



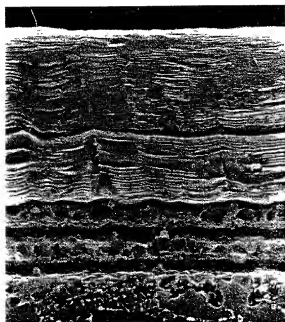


Fig. I Microstructure of the wear-resistant coating on an aircraft engine blade of titanium-based alloy, x500x2.

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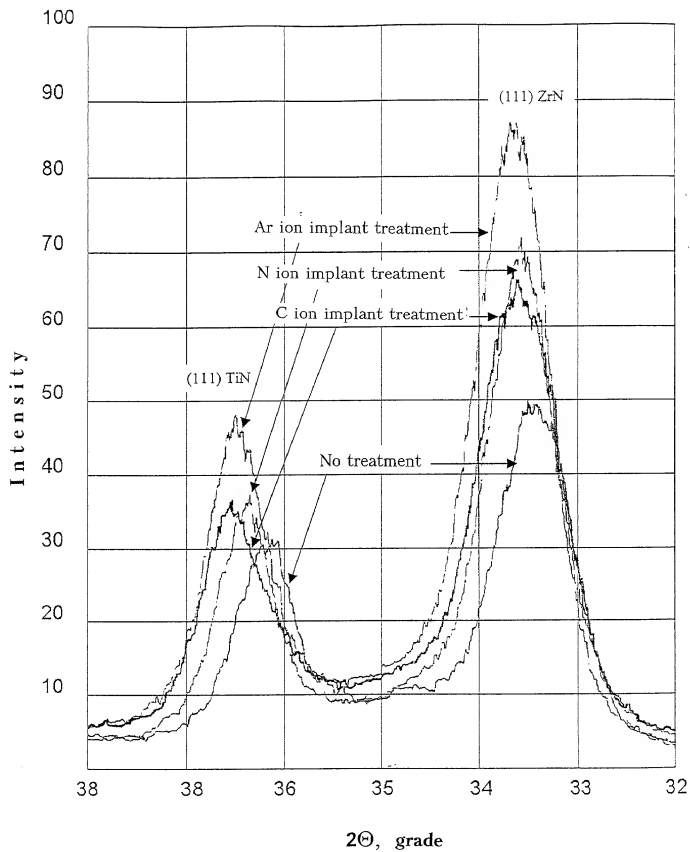
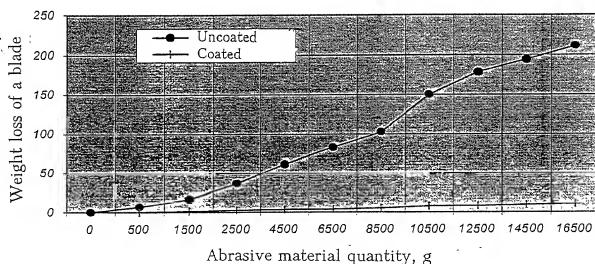


Fig. 2



| Number of test | Abrasive material, weight, g | Weight of blade, g |          | Weight loss, mg |        | Total weight loss, mg |        |
|----------------|------------------------------|--------------------|----------|-----------------|--------|-----------------------|--------|
|                |                              | Uncoated           | Coated   | Uncoated        | Coated | Uncoated              | Coated |
| 0              | 0                            | 25,93467           | 26,68606 | 0               | 0      | 0                     | 0      |
| 1              | 500                          | 25,92766           | 26,68604 | 7,01            | 0,02   | 7,01                  | 0,02   |
| 2              | 1500                         | 25,9176            | 26,68412 | 10,06           | 1,92   | 17,07                 | 1,94   |
| 3              | 2500                         | 25,89843           | 26,68318 | 19,17           | 0,94   | 36,24                 | 2,88   |
| 4              | 4500                         | 25,87291           | 26,68266 | 25,52           | 0,52   | 61,76                 | 3,4    |
| 5              | 6500                         | 25,85201           | 26,68203 | 20,9            | 0,63   | 82,66                 | 4,03   |
| 6              | 8500                         | 25,83202           | 26,68087 | 19,99           | 1,16   | 102,65                | 5,19   |
| 7              | 10500                        | 25,78469           | 26,67814 | 47,33           | 2,73   | 149,98                | 7,92   |
| 8              | 12500                        | 25,75645           | 26,67761 | 28,24           | 0,53   | 178,22                | 8,45   |
| 9              | 14500                        | 25,74019           | 26,6761  | 16,26           | 1,51   | 194,48                | 9,96   |
| 10             | 16500                        | 25,72354           | 26,67494 | 16,65           | 1,16   | 211,13                | 11,12  |

Erosion values represented by weight loss in testing conducted on gas-turbine engine compressor blades



#### Testing conditions:

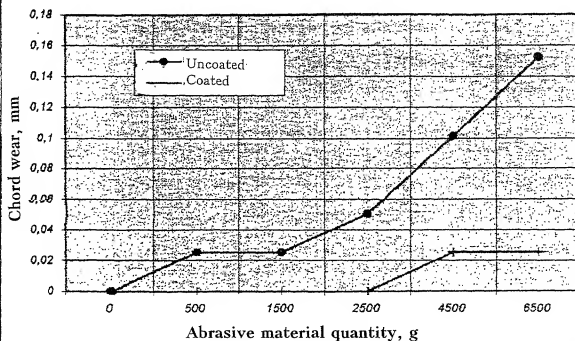
|                    |  |
|--------------------|--|
| Flow rate          | 212 m/s                                |
| Temperature of air | 20 °C                                  |
| Abrasive material  | quartz sand, of 10 microns granularity |
| Attack angle       | 20°                                    |

Fig. 4

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| Number of test | Abrasive material weight, g | Chord    |         | Chord wear, mm |        | Total chord wear, mm |        |
|----------------|-----------------------------|----------|---------|----------------|--------|----------------------|--------|
|                |                             | Uncoated | Coated  | Uncoated       | Coated | Uncoated             | Coated |
| 0              | 0                           | 34,798   | 35,0012 | 0              | 0      | 0                    | 0      |
| 1              | 500                         | 34,7726  | 35,0012 | 0,0254         | 0      | 0,0254               | 0      |
| 2              | 1500                        | 34,7726  | 35,0012 | 0              | 0      | 0,0254               | 0      |
| 3              | 2500                        | 34,7472  | 35,0012 | 0,0254         | 0      | 0,0508               | 0      |
| 4              | 4500                        | 34,8964  | 34,9758 | 0,0508         | 0,0254 | 0,1016               | 0,0254 |
| 5              | 6500                        | 34,8456  | 34,9758 | 0,0508         | 0      | 0,1524               | 0,0254 |

Erosion values represented by chord wear in testing conducted on gas-turbine engine compressor blades



Testing conditions:

|                    |   |
|--------------------|---|
| Flow rate          | 212 m/s                                   |
| Temperature of air | 20 °C                                     |
| Abrasive material  | quartz-sand,<br>of 10 microns granularity |
| Attack angle       | 20°                                       |

Fig. 5

# Fatigue test results obtained on compressor blades with and without coating

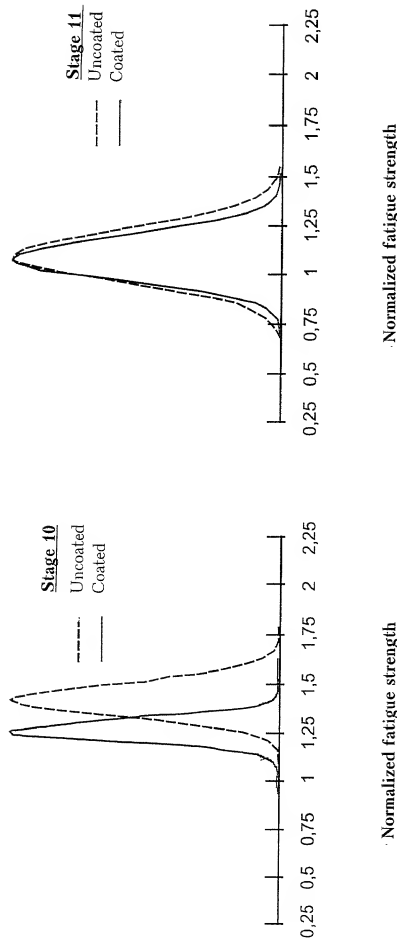


Fig. 6

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

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|--|------------------|---------------------------------------|---------|-----------|
| U.S. APPLICATION NUMBER                      | U.S. FILING DATE | PATENTED                              | PENDING | ABANDONED |
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| PCT APPLICATION NO.                          | PCT FILING DATE  | U.S. SERIAL NUMBERS ASSIGNED (If any) |         |           |
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**POWER OF ATTORNEY:** As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number):

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

|                           |                           |
|---------------------------|---------------------------|
| SIGNATURE OF INVENTOR 101 | SIGNATURE OF INVENTOR 101 |
| ✓ <i>[Signature]</i>      | <i>[Signature]</i>        |
| DATE ✓ 11 September 2000  | DATE 11 September 2000    |

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR DEPOSITION OF WEAR RESISTANT COATINGS TO IMPROVE SERVICE LIFE OF

the specification of which (check only one item below):

COATED COMPONENTS

☐ is attached hereto.

☐ was filed as United States application

Serial No. \_\_\_\_\_

on \_\_\_\_\_

and was amended

on \_\_\_\_\_ (if applicable).

☒ was filed as PCT international application

Number: PCT/RU99/00336

on 14.09.1999

and was amended under PCT Article 19

on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

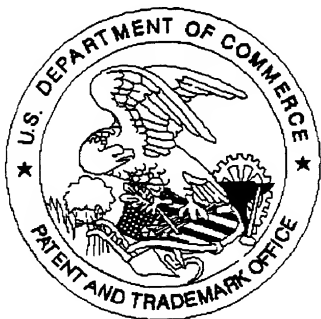
I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

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|-------------------------------------|--------------------|--------------------------------------|---|
| RU                                  | 99118131           | 16.08.1999                           | <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO |
|                                     |                    |                                      | <input type="checkbox"/> YES <input type="checkbox"/> NO            |
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